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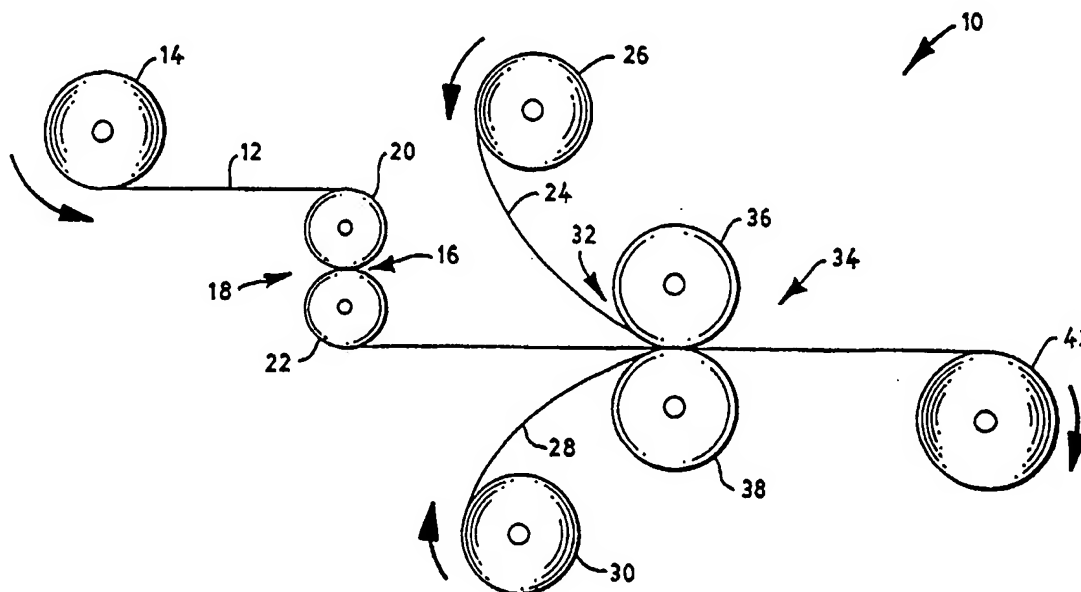
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## Published

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(54) Title: IMPROVED COMPOSITE ELASTIC MATERIAL AND PROCESS FOR PRODUCING THE SAME



## (57) Abstract

The present invention relates to dimensionally stable and/or latent composite elastic laminate materials. The method of manufacturing the composite elastic laminate materials includes providing a polymeric material having a first length, stretching the polymeric material to a second length and bonding at least one non-woven facing to the polymeric material in a calender consisting of two smooth-surfaced rolls, such that the elastic component is not damaged or preconditioned to damage during the use of the personal care article in which the composite elastic material is used.

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## IMPROVED COMPOSITE ELASTIC MATERIAL AND PROCESS FOR PRODUCING THE SAME

### FIELD OF INVENTION

5 The present invention relates to a composite elastic material having improved stress relaxation and improved dimensional stability. This invention also relates to a method of manufacturing composite elastic materials that are dimensionally stable and latent and that have improved stress relaxation.

### BACKGROUND OF THE INVENTION

10 The present invention relates to a dimensionally stable and/or latent composite elastic material having improved stress relaxation and creep resistance and laminates thereof. The present invention also relates to a method of manufacturing the same.

As used herein, the term "composite elastic material" refers to a multicomponent or multilayer elastic material in which one layer is elastic. A composite elastic material that is "dimensionally stable" is one that retains its dimensions, i.e., length and width, under actual use conditions. Use conditions generally involve body temperature, humidity and heat. A "latent elastic laminate material" refers to a laminate material that has an elasticizable component that is dormant but that can be activated at will, normally using a stimulus such as heat. In other words, a latent elastic laminate material will become elastic when activated.

20 The term "stress relaxation" is defined as the load required to hold a constant elongation over a period of time. The term "creep" is defined as the loss of shape or dimension of an article due to some irreversible flow or structural breakdown under a constant load or force. There are two kinds of creep: (1) time-independent, in which the shape changes because of the irreversible flow or structural breakdown under a constant load or force and does not recover when the force is removed; and (2) time-dependent, wherein some of the shape recovers when the force is removed.

Composite elastic materials and laminates thereof have a wide variety of uses, especially in the areas of absorbent articles and disposable items. As used herein, the term "absorbent articles" refers to devices which absorb and contain body exudates and, more specifically, refers to devices that are placed against or in proximity to the body of the wearer to absorb and contain the various exudates discharged from the body. The term "absorbent articles" is intended to include diapers, training pants, absorbent underpants, incontinence products and the like. The term "disposable" is used herein to describe absorbent articles not intended to be laundered or otherwise restored or reused as an absorbent article.

Generally, composite elastic material is a continuous filament-type structure in which a layer of continuous, generally parallel, elastic filaments are bonded to at least one facing layer using a heated calender roll and an anvil roll. Continuous filament laminates are disclosed in U.S. Patent No. 5,366,793 to Fitts, Jr. et al. and U.S. Patent No. 5,385,775 to Wright, both of which are incorporated herein by reference in their entirety.

Typically, the calender roll is patterned in some way so that the resulting laminate material is not bonded across its entire surface. The anvil roll may also be patterned if it is desired. The maximum bond point surface area for a given area of surface on one side of the laminate generally will not exceed about 50% of the total surface area. Typically, the percent bonding area varies from about 10% to about 30% of the area of the laminate material. Such a process is disclosed, for example, in U.S. Patent No. 5,385,775 to Wright and U.S. Patent No. 4,041,203 to Brock et al., both of which are incorporated herein by reference in their entirety.

One disadvantage to this method of lamination is that the patterned rolls severely damage the elastic filaments. The damage to the elastic filaments affects the elastic properties and, thus, the performance of the composite elastic material and laminates thereof by causing the fibers to break during use, at body temperature, and under stretched conditions.

A need, therefore, exists for a method of manufacturing a composite elastic material that is dimensionally stable. Additionally, there is a need for a method of manufacturing a composite elastic material without damaging the polymer strands during the manufacturing process.

## 5 SUMMARY OF THE INVENTION

The present invention provides a process for producing a dimensionally stable and/or latent elastic laminate material. The process includes the steps of providing a polymeric material having a first length, stretching the polymeric material to a second length and bonding at least one  
10 non-woven facing to the polymeric material in a calender having two smooth-surfaced rolls in order to reduce damage to the structure of the elastic material. The present invention also provides a process wherein 100% of the surface area of the roll contacts the elastic material. The present invention, therefore, produces a single composite elastic material having minimal to  
15 negligible damage to the polymeric material by calendering a polymeric material and at least one non-woven facing between smooth-surfaced rolls.

The process of the present invention not only overcomes the problems of the prior art, but also provides several advantages. These include: (1) a substantial improvement in the performance of the resulting laminate via  
20 dimensional stability; (2) a reduced load loss over time under actual use conditions; (3) a possible cost reduction through the use of a lower amount of elastic material in the final laminate; (4) the production of latent and/or heat shrinkable materials; and (5) the use in the resulting laminate of the full potential of the elastic polymer. The present invention also provides a  
25 method of manufacturing a low cost elastic non-woven material.

Composite elastic laminate materials produced according to the present invention may be used as elastic components of personal care absorbent articles such as, for example, in the side panels of diapers and training pants. They may also be used in the leg elastic and gasketing of  
30 diapers, training pants, incontinence devices and the like.

The foregoing and other features and advantages of the present invention will become apparent from the following detailed description of the presently preferred embodiments, when read in conjunction with the accompanying examples.

5      **BRIEF DESCRIPTION OF THE DRAWINGS**

FIG. 1 is a schematic drawing of the process for forming a composite elastic material according to the present invention.

10      FIG. 2 is a perspective view of an exemplary disposable garment, in this case training pants, that utilizes the laminate material made according to the present invention.

FIG. 3 is a graph of stress relaxation modulus versus time determined during stress relaxation testing of a composite elastic laminate material produced using a pattern roll and testing of a composite elastic laminate material produced using two smooth rolls.

15      **DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS**

20      The present invention is directed to a dimensionally stable composite elastic material and laminates thereof having improved stress relaxation and improved creep resistance. The present invention also relates to a method for forming composite elastic laminate materials that are dimensionally stable and latent and that have improved stress relaxation properties.

Referring now to the drawings wherein like reference numerals represent the same or equivalent structure and, in particular, to FIG. 1 of the drawings, there is illustrated at **10** a process for forming a composite elastic material using smooth-roll calendering.

25      According to the present invention, an elastic web **12** is unwound from a supply roll **14** and travels in the direction indicated by the arrow associated therewith as the supply roll **14** rotates in the direction of the arrows associated therewith. The physical structure of the elastic web **12** can be a

film, a non-woven or strands. The elastic web 12 passes through the nip 16 of the S-roll arrangement formed by the stack rollers 20 and 22.

The elastic web 12 may also be formed in a continuous process such as, for example, the process described below, and passed through the nip 16 without being stored on a supply roll.

A first gatherable layer 24 is unwound from a supply roll 26 and travels in the direction indicated by the arrow associated therewith as the supply roll 26 rotates in the direction of the arrows associated therewith. A second gatherable layer 28 is unwound from a second supply roll 30 and travels in the direction indicated by the arrow associated therewith as the supply roll 30 rotates in the direction of the arrows associated therewith. The first gatherable layer 24 and the second gatherable layer 28 pass through the nip 32 of the calender roll arrangement 34 formed by the calender roll 36 and 38. The first gatherable layer 24 and/or the second gatherable layer 28 may be formed by extrusion processes such as, for example, meltblowing processes, spunbonding processes or film extrusion processes and passed directly through the nip 32 without first being stored on a supply roll.

The elastic web 12 passes through the nip 16 of the S-roll arrangement 18 in a reverse-S path as indicated by the rotation direction arrows associated with the stack rollers 20 and 22. From the S-roll arrangement 18, the elastic web 12 passes through the pressure nip 32 formed by the calender roll arrangement 34. Additional S-roll arrangements (not shown) may be introduced between the S-roll arrangement 18 and the calender roll arrangement 34 to stabilize the stretched material and to control the amount of stretching. Because the peripheral linear speed of the rollers of the S-roll arrangement 18 is controlled so as to be less than the peripheral linear speed of the rollers of the calender roll arrangement 34, the elastic web 12 is tensioned between the S-roll arrangement 18 and the pressure nip of the calender roll arrangement 34. Importantly, filaments of the elastic web 12 in strand form should run along in the direction that the film is stretched so

that the filaments can provide the desired stretch properties in the finished composite material. By adjusting the difference in the speeds of the rollers, the elastic web **12** is tensioned so that it stretches a desired amount and is maintained in such stretched condition while the first gatherable layer **24** and the second gatherable layer **28** are joined to the elastic web **12** during their passage through calender rolls **36** and **38** to form a composite elastic material **40**. The surfaces of calender rolls **36** and **38** are smooth and patternless. Thus, the bond area of the composite elastic material **40** is 100% because both of the calender rolls are smooth-surfaced.

Preferably, the stretched length of the polymeric material is at least about 50% of the original length. Preferably, the stretched length is up to about 95% of the ultimate elongation of the elastomer. It should be noted that different elastomers have different elongations. Even more preferably, the stretched length should be in the range of about 200% to about 600% where percent elongation is defined according to the following formula:

$$\frac{\text{final length} - \text{initial length}}{\text{initial length}} \times 100\%$$

Referring again to FIG. 1, the composite elastic material **40** immediately relaxes upon release of the tensioning force provided by the S-roll arrangement **18** and the bonder roll arrangement **34**, whereby the first gatherable layer **24** and the second gatherable layer **28** are gathered in the composite elastic material **40**. Preferably, a permanent elongation length of from about 1.5 times the original length is retained after the stretched film is allowed to relax.

The composite elastic material **40** is then wound up on a winder **42**. The composite elastic material **40** may be wound under tension or without tension. If wound without tension, the composite elastic material will be stored in the roll in its unstretched state such that the material may be stretched at any time. If wound with tension, the composite elastic material is



stored in a stretched condition. The elastic properties of the material stored in the stretched condition can be reactivated using heat or other conditions. Alternatively, the composite elastic material 40 may continue in line for further processing or conversion (not shown).

5           Polymeric materials that are useful in forming the elastic web 12 are generally known as "elastomers." An elastomer is a rubber elastic material capable of stretching to several times its original, relaxed length and which tends to recover completely its elongation upon release of the stretching, biasing force. As used herein, the term "recover" refers to a contraction of a stretched material upon termination of a biasing force following the stretching of the material by application of the biasing force. Examples of these materials are indexed as "elastomers" in Bradley et al., Materials Handbook, 284-290 (McGraw-Hill, Inc. 1991), incorporated herein by reference.

10           The elastomers useful in the present invention may be selected from the group consisting of elastomeric thermoplastic polymers. The physical structure of the elastomer can be strands, cast or blown film, crimped, any non-woven web of fiber of a desired thermoplastic polymer or a combination thereof.

15           Suitable elastomeric thermoplastic polymers include styrene block copolymers such as, for example, those available under the trademark KRATON® from Shell Chemical Company of Houston, Texas. KRATON® block copolymers are available in several different formulations, a number of which are identified in U.S. Patents 4,663,220; 4,323,534; 4,834,738; 5,093,422; and 5,304,599 which are incorporated herein by reference.

20           Other exemplary elastomeric materials that may be used include polyurethane elastomeric materials such as, for example, those available under the trademark PELLATHANE® from Dow Chemical Company of Midland, Michigan or under the trademark ESTANE® from B.F. Goodrich & Company of Akron, Ohio or under the trademark MORTHANE® from Morton Thiokol Corporation; polyester elastomeric materials such as, for example, those available under the trade designation HYTREL from E.I Dupont de

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Nemours & Company of Wilmington, Delaware and those known as ARNITEL® which were formerly available from Akzo Plastics of Arnhem, Holland and now available from DSM of Sittard, Holland; and polymers from metallocene-based catalysis which are available under the name ENGAGE® from Dow Chemical Company of Midland, Michigan for polyethylene-based polymers and from Exxon Chemical Company of Baytown, Texas under the trade name ACHIEVE® for polypropylene-based polymers and EXACT® and EXCEED® for polyethylene-based polymers.

The gatherable layers, or facings, 24 and 28 may be fibrous non-woven materials such as, for example, spunbonded webs or meltblown webs. A non-woven web, as described herein, means a web having a structure of individual fibers or threads that are interlaid, but not in an identifiable, repeating manner. A plurality of fibrous non-woven facings 24 and 28, as shown in FIG. 1, may also be used depending on the basis weight of the non-woven material used.

The gatherable layers 24 and 28 may be formed by a variety of processes including, but not limited to, meltblowing and spunbonding processes. Meltblown fibers are fibers formed by extruding a molten thermoplastic material through a plurality of fine, usually circular, capillaries of a meltblowing die as molten threads or filaments into converging high-velocity, usually hot, gas (e.g., air) streams which are flowing in the same direction as the extruded filaments or threads of the molten thermoplastic material so that the extruded filaments or threads are attenuated, i.e., drawn or extended, to reduce their diameter. The threads or filaments may be attenuated to microfiber diameter which means the threads or filaments have an average diameter not greater than about 75 microns, generally from about 0.5 microns to about 50 microns, and more particularly from about 2 microns to about 40 microns. Thereafter, the meltblown fibers are carried by the high-velocity gas stream and are deposited on a collecting surface to form a web of randomly disbursed meltblown fibers. The meltblown process is well-known and is described in various patents and publications, including NRL

Report 4364, "Manufacture of Super-Fine Organic Fibers" by B.A. Wendt, E.L. Boone and D.D. Fluharty; NRL Report 5265, "An Improved Device for the Formation of Super-Fine Thermoplastic Fibers" by K.D. Lawrence, R.T. Lukas and J.A. Young; U.S. Patent No. 3,676,242 to Prentice; and U.S. Patent No. 3,849,241 to Buntin et al. The foregoing references are incorporated herein in by reference in their entirety. Meltblown fibers are microfibers which may be continuous or discontinuous, are generally smaller than 10 microns in average diameter and are generally tacky when deposited onto a collecting surface.

Spunbonded fibers are small diameter fibers that are formed by extruding a molten thermoplastic material as filaments from a plurality of fine, usually circular, capillaries of a spinneret with the diameter of the extruded filaments then being rapidly reduced as by, for example, non-eductive or eductive fluid-drawing or other well-known spunbonding mechanisms. The production of spunbonded non-woven webs is illustrated in patents such as, for example, U.S. Patent No. 4,340,563 to Appel et al.; U.S. Patent No. 3,802,817 to Matsuki et al.; U.S. Patent No. 3,692,618 to Dorschner et al; U.S. Patent No. 3,542,615 to Dobo; U.S. Patent No. 3,502,763 to Hartman; U.S. Patent No. 3,502,538 to Peterson; U.S. Patent Nos. 3,341,394 and 3,338,992 to Kinney; U.S. Patent No. 3,276,944 to Levy; and Canadian Patent No. 803,714 to Harmon. The disclosures of these patents are herein incorporated by reference in their entirety. Spunbonded fibers generally are not tacky when deposited onto a collecting surface. Spunbonded fibers generally are continuous and have average diameters (from a sample of at least 10) larger than 7 microns and, more particularly, from about 10 microns to about 20 microns.

Preferably, the non-woven gatherable layers comprise spunbonded fibers. For most uses, the total weight of the spunbonded facing material is about 0.4 ounces per square yard.

Various techniques may be employed to secure the elastic web onto the non-woven facings such as, for example, adhesive

bonding. In adhesive bonding, an adhesive such as a hot melt, pressure sensitive adhesive is applied between the polymeric material and the facing to bind the polymeric material and facing together. The adhesive can be applied by, for example, melt spraying, printing or meltblowing. Various types of adhesives are available, including those produced from amorphous polyalphaolefins, ethylene vinyl acetate-based hot melts and KRATON® brand adhesives available from Shell Chemical Company of Houston, Texas.

The resulting composite elastic material **40** has elastic properties identical to those of the pure polymeric material. The resulting laminate material suffers no loss of elasticity and provides better body conformance than laminate materials produced using one or more patterned calender rolls. The filaments in the laminate materials of the present invention are also less likely to break.

The stress relaxation of the resulting composite elastic material **40** is at least about 1 psi less than the stress relaxation of a thermal, pattern-bonded material of similar composition at a time of about 8 hours. Preferably, the stress relaxation is at least about 2 psi less than that of a pattern-bonded material at a time of about 8 hours.

Referring now to FIG. 2, there is illustrated a disposable garment **50** incorporating an elastic laminate made according to the present invention. Although training pants are shown in FIG. 2, it will be understood that use of the elastic laminate produced according to the present invention is not limited to such articles and may also be used in a wide variety of applications including, but not limited to, diapers, incontinence devices and the like.

Referring again to FIG. 2, the disposable garment **50** includes waste containment section **52** and two side panels **54** and **56** defining a waist opening **58** and a pair of legs openings **60** and **62**. FIG. 2 illustrates the disposable garment **50** fitted on a wearer's torso **64** in dashed lines. Side panel **54** includes stretchable side member **66** and stretchable side member **68** connecting intermediate member **70** which is made of a non-stretchable

material. Similarly, side panel 56 includes stretchable side member 72 and stretchable side member 74 connecting intermediate member 76 which is made of a non-stretchable material. Disposable garment 50 also includes front waist elastic member 78 and rear waist elastic member 80 for providing additional elasticity along waist opening 58. Leg elastics 82 are provided with waist containment section 52 between side panels 54 and 56.

The composite elastic material of the present invention may be used to form various portions of the disposable garment 50 and particularly, the side panels 54 and 56. The laminate material may also be used in the leg elastics 82 of the disposable garment 50.

### EXAMPLES

A composite elastic material was made using two smooth-surfaced calender rolls according to the present invention. A control material was made using pattern rolls according to the prior art process. Both materials were made from the same polymer blend.

#### Stress Relaxation

Stress relaxation of the control and inventive samples was measured on a Sintech 1/S tensile test frame available from Sintech, Inc. of Stoughton, Massachusetts. Sample size was about 3 inches wide and 7 inches long. Each specimen was clamped between the jaws of the grip at a 3-inch grip-to-grip distance. Each sample and the grip fixtures were enclosed in an environmental chamber and equilibrated at 100°F for 3 minutes. Each sample was then stretched to a final constant elongation of 4.5 inches (50% elongation) at a cross-head displacement of 20 inches per minute. The load required to maintain the 50% elongation as a function of time was monitored for 8 hours for each sample.

Data from the Sintech 1/S system was reduced by calculating the engineering stress (pounds per square inch, or psi) from a knowledge of the initial cross-sectional area of each sample. Strain, or elongation, was calculated from the initial grip-to-grip distance and the constant elongation.

The ratio of the stress and strain gives the stress relaxation modulus (psi). This data was used to generate stress relaxation modulus versus time curves for the control and inventive composite elastic laminate materials. FIG. 3 is a graph showing the stress relaxation modulus versus time curves of the composite elastic laminate material produced using a pattern roll (A) and the composite elastic laminate material produced using two smooth rolls (B).

The resulting data can be fitted to the following power-law model to obtain the exponent, m:

$$\sigma = (\sigma_{t=0.1 \text{ min.}})(t^{-m}),$$

wherein  $\sigma$  is stress,  $t$  is time and  $m$  represents how fast the material loses its load, or elastic properties. Table I shows the rate of actual load loss, or slope, as calculated using the above-described formula and the actual load loss in 8 hours at 100°F. As can be seen in the Table, the use of the smooth calender rolls decreases the magnitude of the slope and the percent load loss favorably.

TABLE I

Sample ID	Slope	% Load Loss
Pattern Roll	-0.13	68%
Smooth Rolls	-0.10	55%

Of course, it should be understood that a wide range of changes and modifications can be made to the embodiments described above. It is, therefore, intended that the foregoing description illustrate rather than limit this invention and that it is the following claims, including all equivalents, that define this invention.

**CLAIMS:**

1. A process for producing dimensionally stable composite elastic laminate materials comprising:

providing a polymeric material having a first length;

5 stretching said polymeric material to a second length; and

bonding at least one facing to said polymeric material in a calender consisting of two smooth-surfaced rolls.

2. The process of claim 1 wherein said polymeric material is selected from the group consisting of elastomeric thermoplastic polymers.

10 3. The process according to claim 2 wherein said elastomeric thermoplastic polymer is selected from the group comprising block copolymers, polyurethanes, polyesters and polymers from metallocene-based catalysis.

15 4. The process of claim 3 wherein said elastomeric thermoplastic polymer is a block copolymer.

5. The process of claim 3 wherein said elastomeric thermoplastic polymer is polyurethane.

6. The process of claim 3 wherein said elastomeric thermoplastic polymer is polyester.

20 7. The process of claim 3 wherein said elastomeric thermoplastic polymer is a polymer from metallocene-based catalysis.

8. The process of claim 1 wherein said second length of said polymeric material is at least 50% of said first length.

9. The process of claim 1 wherein the percent elongation of said polymeric material is from about 200% to about 600%.

10. The process of claim 1 wherein said facing is a fibrous non-woven web.

5 11. The process of claim 10 wherein said fibrous non-woven web comprises a web of meltblown fibers.

12. The process of claim 10 wherein said fibrous non-woven web comprises a web of spunbonded fibers.

10 13. The process according to claim 12 wherein the basis weight of said fibrous non-woven web is about 0.4 ounces per square yard.

14. The process of claim 1 wherein said polymeric material is bonded to said facing with an adhesive.

15. A process of producing dimensionally stable and/or latent elastic laminate materials comprising:

15 providing a polymeric material having a first length;

stretching said polymeric material to a second length;

applying an adhesive to a facing; and

bonding said facing to said polymeric material in a calender consisting of two smooth-surfaced rolls.

20 16. The process of claim 15 wherein said adhesive is a hot melt, pressure sensitive adhesive.

17. The process of claim 15 wherein said polymeric material is selected from the group consisting of elastomeric thermoplastic polymers.



18. The process of claim 17 wherein said of elastomeric thermoplastic polymer is selected from the group comprising block copolymers, polyurethanes, polyesters and polymers from metallocene-based catalysis.

5 19. The process of claim 17 wherein said facing is a fibrous non-woven web.

20. The process of claim 19 wherein said fibrous non-woven web comprises a web of meltblown fibers.

10 21. The process of claim 19 wherein said fibrous non-woven web comprises a web of spunbonded fibers.

15 22. A dimensionally stable composite elastic material comprising a polymeric material and a fibrous, non-woven facing, wherein said composite elastic material has a stress relaxation of at least about 1 psi less than the stress relaxation of a pattern bonded material of similar composition at a time of about eight hours.

23. The dimensionally stable composite elastic material of claim 22 wherein said composite elastic material has a stress relaxation of at least about 2 psi less than the stress relaxation of a pattern bonded material of similar composition at a time of about eight hours.

20 24. A disposable personal care absorbent article comprising a liquid permeable liner and an outer cover with an absorbent core disposed therebetween, wherein said outer cover is formed such that the stress relaxation is at least about 1 psi less than the stress relaxation of a pattern bonded material of similar composition at a time of about eight hours.

25 25. The disposable absorbent article of claim 24 wherein the outer cover of said absorbent article is formed such that the stress relaxation is at

least about 2 psi less than the stress relaxation of a pattern bonded material of similar composition at a time of about eight hours.

26. The disposable absorbent article of claim 24 wherein said article is a diaper.

5           27. The disposable absorbent article of claim 24 wherein said article is a training pant.

28. The disposable absorbent article of claim 24 wherein said article is an adult incontinence garment.

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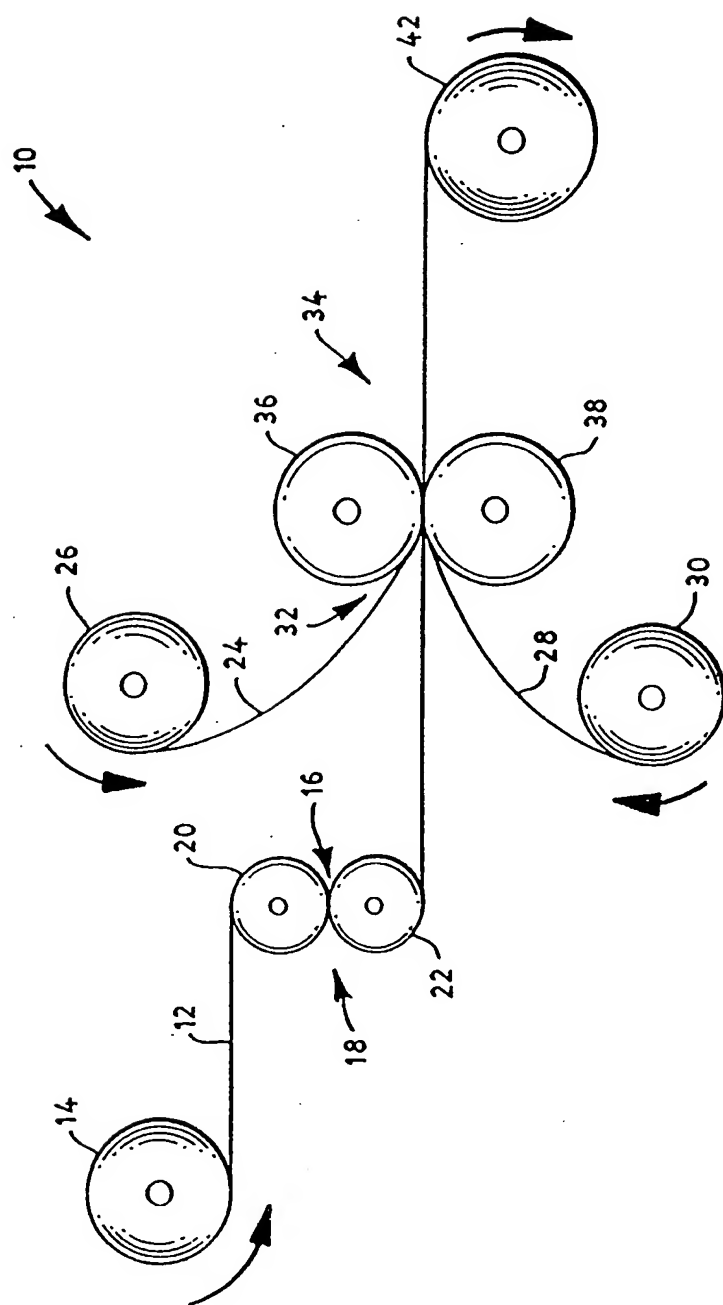


FIG. 1

**2/3**

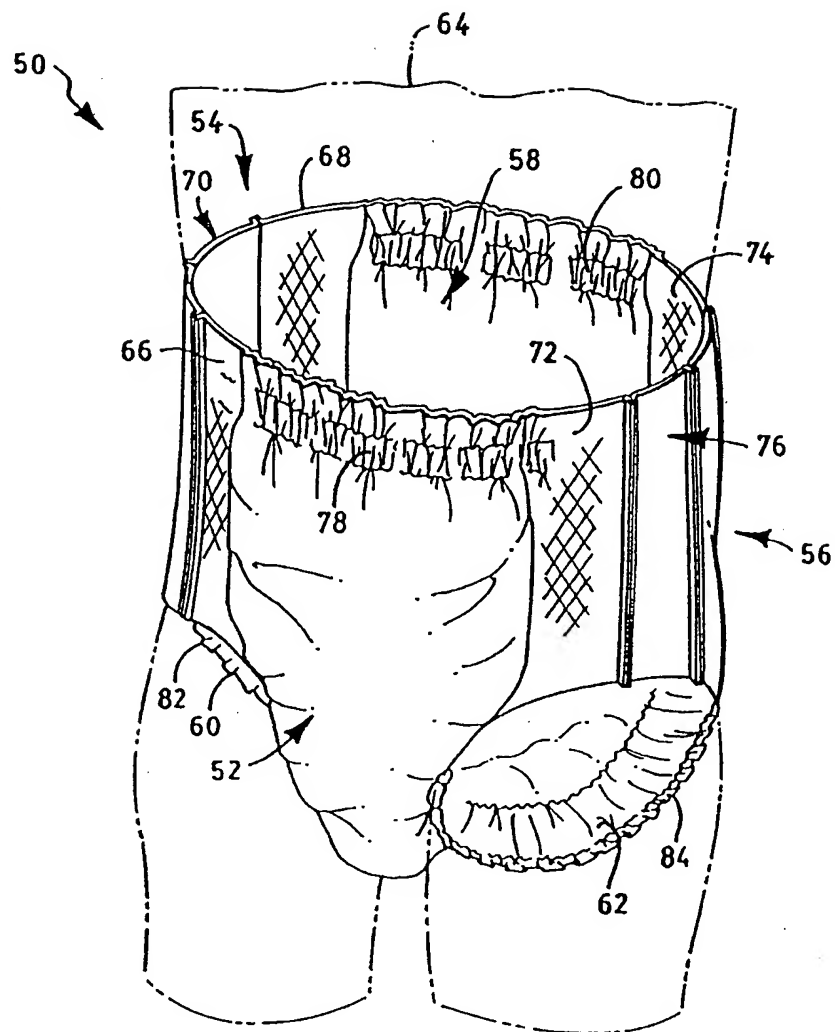


FIG. 2

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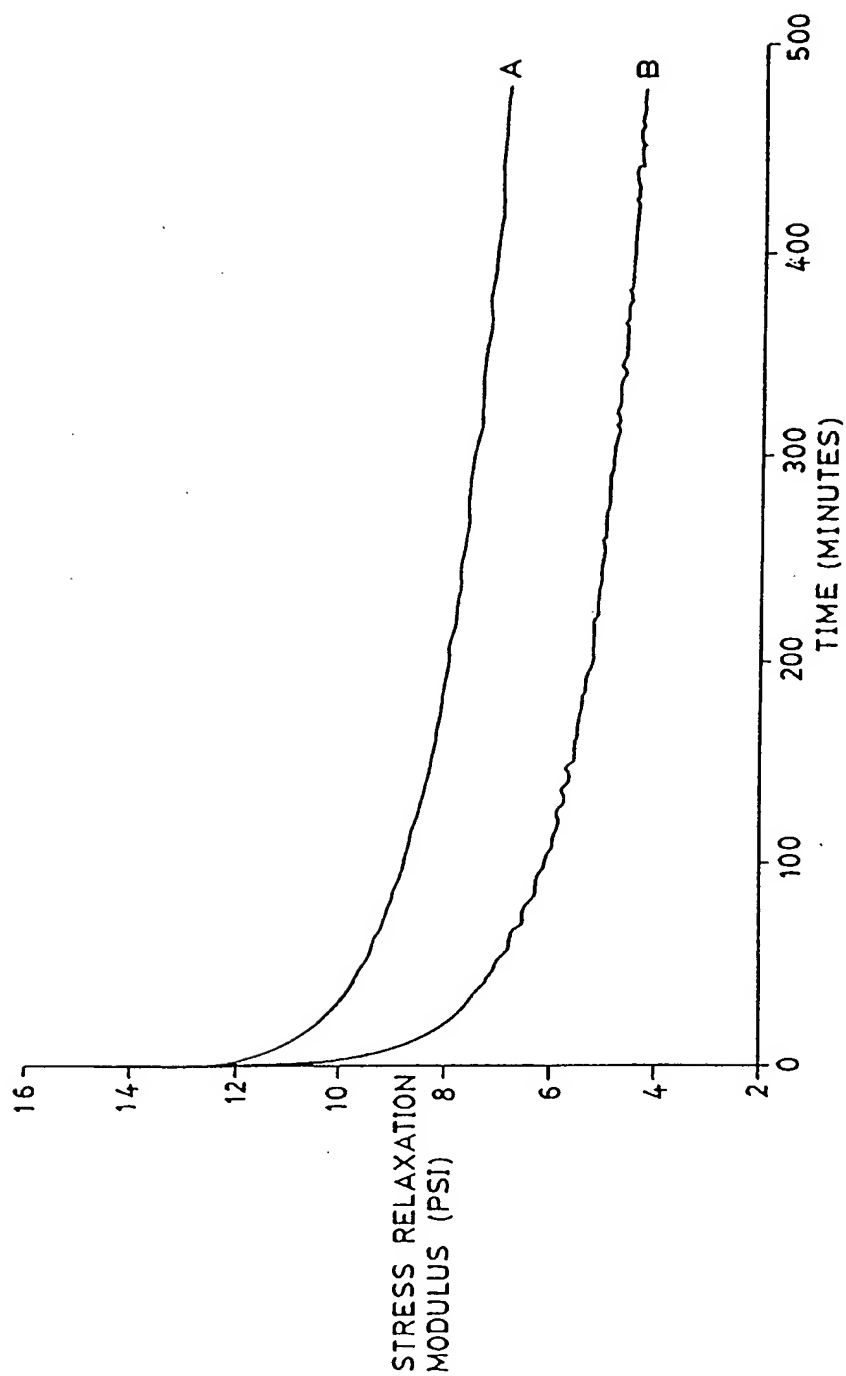


FIG. 3

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 97/23825

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 B32B31/12 B32B5/02 B32B7/04 A61F13/15

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 B32B A61F D04H B29C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 90 03464 A (KIMBERLY-CLARK CORPORATION) 5 April 1990 see page 5, line 6 - line 23 see page 14, line 28 - line 30; claims 1-9, 11-14; figure 1 ---	1-28
X	EP 0 330 716 A (HENNING J. CLAASSEN) 6 September 1989 see the whole document ---	1-5, 8-28
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

16 April 1998

Date of mailing of the international search report

23/04/1998

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# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 97/23825

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A	EP 0 327 402 A (TONEN SEKIYUKAGAKU KABUSHIKI KAISHA) 9 August 1989 see page 6, line 44 - line 54; figures 1,2 ---	1
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A	US 4 259 220 A (WILLIAM L. BUNNELLE) 31 March 1981 see claim 1; figure 1 -----	16

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